

## MICROREACTOR

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**Applicant:** SEIKO EPSON CORP

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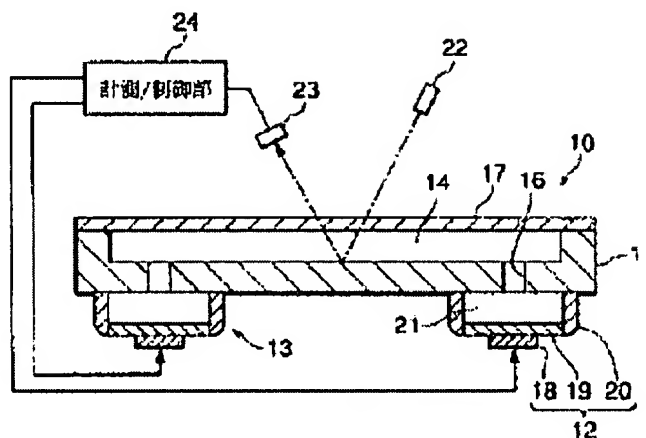
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### Abstract of JP2001228159

**PROBLEM TO BE SOLVED:** To realize the liquid delivery to a channel in a microreactor with high precision. **SOLUTION:** The capacity of a cavity 21 formed on the inner wall of an injection port 12 is changed by the drive of a piezoelectric element 18 to deliver and receive a liquid to and from a channel 14. Since the piezoelectric element 18 can deliver and receive the liquid to and from the channel 14 with a resolution of 0.5 pl to 60 pl, precise handling of a molecule can be realized by the minute drive of the piezoelectric element 18.



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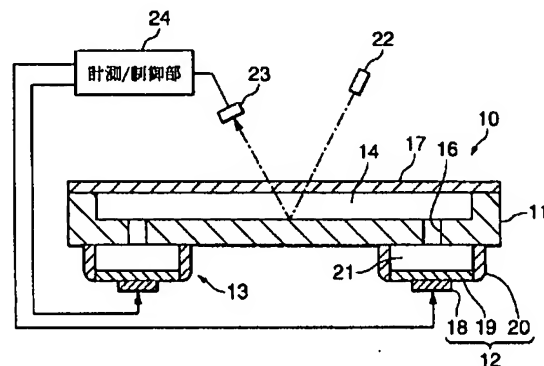
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(54) 【発明の名称】 マイクロリアクタ

(57) 【要約】

【課題】 マイクロリアクタにおけるチャネルへの送液を精密な精度で実現する。

【解決手段】 圧電体素子(18)の駆動により注入ポート(12)の内壁に形成されたキャビティ(21)の容積を変化させ、チャネル(14)との間で試料を送受液する。圧電体素子(18)は0.5p l乃至60p lの分解能でチャネル(14)へ送受液することができるため、圧電体素子(18)の微細な駆動によって精密な精度で分子のハンドリングを実現することができる。



【特許請求の範囲】

【請求項1】 インクジェット方式によってチャネルとの間で試料を送受液する注入ポートと、当該チャネルを介して注入ポートに連通し、化学反応後の試料を回収するための排出ポートを備えたマイクロリアクタ。

【請求項2】 試料の化学反応を検出する検出器と、検出したデータに基づいて、注入ポートからチャネルへの試料の送受液量を調整する制御部とをさらに備えた請求項1に記載のマイクロリアクタ。

【請求項3】 前記検出器は試料の化学反応を光学的に検出する請求項2に記載のマイクロリアクタ。

【請求項4】 圧電体素子の駆動によりキャビティの容積を変化させ、チャネルとの間で試料を送受液する注入ポートと、当該チャネルを介して注入ポートに連通し、化学反応後の試料を回収するための排出ポートを備えたマイクロリアクタ。

【請求項5】 試料の化学反応を検出する検出器と、検出したデータに基づいて圧電体素子の駆動を制御し、試料の送受液量を調整する制御部とをさらに備えた請求項4に記載のマイクロリアクタ。

【請求項6】 前記検出器は試料の化学反応を光学的に検出する請求項5に記載のマイクロリアクタ。

【請求項7】 前記注入ポートはタンクに貯蔵された試料を圧電体素子の駆動によってキャビティ内へ導入し、さらにチャネルへ送液する請求項4乃至請求項6のうち何れか1項に記載のマイクロリアクタ。

【請求項8】 前記圧電体素子は0.5 p l乃至60 p lの分解能でチャネルとの間で送受液する請求項4乃至請求項7のうち何れか1項に記載のマイクロリアクタ。

【請求項9】 液体吐出ヘッドを用いてマイクロリアクタの注入ポートのキャビティに試料を充填する試料充填方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】 本発明は化学反応を行うためのマイクロリアクタの改良技術に関する。

【0002】

【従来の技術】 特開平10-337173号公報には多数の生化学反応を並列的に行うためにマイクロリアクタを利用する技術が提案されている。マイクロリアクタはマイクロ領域での化学反応実験、薬品の開発、人工臓器開発、ゲノム・DNA解析ツール、マイクロ流体工学の基礎解析ツール等に利用されている。このマイクロリアクタを用いる化学反応にはフラスコを用いた通常の化学反応にはない特徴がある。例えば、装置全体が小さいために熱交換率が極めて高く、温度制御が効率良く行える利点がある。この利点を利用すれば、精密な温度制御を必要とする反応や急激な加熱又は冷却を必要とする反応でも、マイクロリアクタを利用すれば容易に行うことが可能となる。

【0003】 また、微小空間の中で反応を行うため、例えば、有機溶媒と水との液・液界面や、液体と器壁との固・液界面では、いずれの場合も液体の体積に比べて界面の面積の割合が非常に大きい。それ故、分子の移動速度が速く、不均一反応を効率良く行うことができる。さらに、リアクタ（反応槽）の容積は微小であるため、反応に用いる試料（反応試薬、サンプル等）の量及びコストを抑えることができ、生成物の分析能力の限界まで反応スケールを小さくすることで環境への影響を小さくすることができる。

【0004】

【発明が解決しようとする課題】 ところで、マイクロリアクタではチャネル（流路）やリアクタの容積が微小であるため、通常の反応容器とは異なる分子のハンドリングが要求される。従来のマイクロリアクタではシリンジポンプ或いはダイヤフラム型のマイクロポンプを用いてチャネルやリアクタ内に試料を送液していたが、微小且つ複雑なチャネルをもつマイクロリアクタにおいては上記の送液法（pressure pumping）では正確な送液制御が困難であった。

【0005】 このため、電気浸透法による送液法（electroosmotic pumping）が利用されているが、この方法では界面に電気二重層を形成することが困難な有機溶媒には不向きである。H.SalimiMoosavi, T.Tang, J.Am.Chem.Soc. 119, 8716 (1997) には、有機溶媒に電界質を添加して電気浸透法を用いた実験結果が報告されている。このように、マイクロリアクタにおいては、精密な分子のハンドリングを実現することが重要な技術的課題となっている。

【0006】 そこで、本発明はチャネルへの送受液を精密な精度で可能にした注入ポートを備えるマイクロリアクタを提供することを課題とする。また、注入ポートのキャビティ内への試料の注入を精密な精度で充填する試料充填方法を提供することを課題とする。

【0007】

【課題を解決するための手段】 上記の課題を解決するため、本発明のマイクロリアクタはインクジェット方式によってチャネルとの間で試料を送受液する注入ポートと、当該チャネルを介して注入ポートに連通し、化学反応後の試料を回収するための排出ポートを備えて構成されている。インクジェット方式による試料の送受液には、例えば、圧電体素子の機械的変位により注入ポートの内壁に形成されたキャビティの容積を変化させ、キャビティ内の試料を送液するピエゾジェット方式であってもよく、また、熱の印加により急激に蒸気が発生することにより試料を送液するバブルジェット方式であってもよい。

【0008】 圧電体素子を駆動源とするピエゾジェット方式であれば、0.5 p l乃至60 p lの分解能でチャネルとの間で送受液することができるため、注入ポート

とチャネル間の送受液を精密な精度で実現することができる。

【0009】上記の構成においてさらに、試料の化学反応を検出する検出器と、検出したデータに基づいて注入ポートからチャネルへの送受液量を調整する制御部とを備えてもよい。このように構成することで、試料の化学反応の状態に応じて送受液量をフィードバック制御することができる。この場合、化学反応の検出手段として、光学的に検出する手段が好適である。マイクロ領域での検出に優れているためである。

【0010】注入ポートのキャビティ内へ試料を供給するための構成として、タンクに貯蔵された試料を圧電体素子の駆動によってキャビティ内へ導入し、さらにチャネルへ送液するように構成してもよい。

【0011】また、本発明の試料充填方法では液体吐出ヘッドを用いてマイクロリアクタの注入ポートのキャビティに試料を充填する。

【0012】

【発明の実施の形態】以下、各図を参照して本実施の形態について説明する。

【0013】図1にマイクロリアクタ10の模式的な概念図を示す。マイクロリアクタ10はシリコン基板11を微細加工することで製造することができ、注入ポート12、13、チャネル14、及び排出ポート15を備えて構成されている。チャネル14は注入ポート12、13、及び排出ポート15を相互に連通する流路であり、T字型リアクタを構成する。注入ポート12、13に注入された試料はチャネル14を介して合流し、当該合流箇所からチャネル14の下流側がリアクタとして機能する。以下、チャネル14のうち化学反応が生じている部分をリアクタという。反応後の試料は排出ポート15から回収される。尚、説明を簡略するため、同図においては、後述する半導体レーザ22、光センサ23、及び計測/制御部24等は図示していない。

【0014】図2は図1のA-A断面図である。シリコン基板11の表面に形成されたチャネル14上には平板17が貼り合わされており、チャネル14を密封している。また、注入ポート12、13からチャネル14内へ試料を供給できるように、各ポートに対応する位置には貫通孔（オリフィス）16が開口している。注入ポート12、13は圧電体素子18、振動板19、及び側壁20から構成されている。注入ポート12、13の内壁とシリコン基板11によって、試料を充填するためのキャビティ21が形成される。圧電体素子18は電気エネルギーを機械エネルギーに変換する素子であり、逆圧電効果によって変位する圧電性セラミックス、例えば、ジルコン酸チタン酸鉛（PZT）を備えている。この圧電セラミックスに電圧を印加すると、圧電体素子18は膜厚方向に膨張するとともに、幅方向に収縮する。この収縮によって圧電体素子18と振動板19の界面に圧縮応力

が働き、キャビティ21の容積が変化する。この結果、キャビティ21内の試料が貫通孔16を介してチャネル14へ送液可能となる。圧電体素子18は数MHzの振動周波数で駆動することができるため、精密な送受液制御に好適である。振動板19はシリコン酸化膜、シリコン窒化膜等の絶縁膜で構成されている。

【0015】平板17は耐熱ガラスのような透明基板から構成されており、リアクタにおける化学反応を光学的に検出することができる。リアクタにおける化学反応の光学的検出手段として、例えば、分光法を利用することができる。同図において、半導体レーザ22から射出した入射光は平板17及びリアクタ内の反応生成物を透過し、リアクタの壁面で反射した後に光センサ23によって検出される。計測/制御部24は入射光の強度と反射光の強度を測定して、リアクタ内の反応生成物を定量する。計測/制御部24は注入ポート12、13における圧電体素子18の駆動制御回路を備えており、リアクタ内の反応生成物を参照して注入ポート12、13における試料の送受液量を0.5p1～60p1の範囲で調整可能に構成されている。

【0016】尚、リアクタにおける化学反応の検出方法として、上記の他にレーザ蛍光検出器を用いてもよい。レーザは対物レンズを用いることで効率良く集光することができ、高感度の検出を行うことができる。また、マイクロリアクタの全面にレーザ光を照射し、試料の注入、化学反応、分離の様子を高感度カメラで撮影することもできる。電気化学検出器や質量分析装置では出口における試料の分析しか行えないが、このような方式ではマイクロリアクタ全体の現象を観察することができる。また、試料の検出に蛍光法や吸光法を利用する場合にはアダマール変換電気泳動分析を併用すると効果的である。

【0017】また、反応条件が異なるマイクロリアクタ10を多数配置し、高感度フィルムやCCDカメラを用いて多数のリアクタ内での反応を同時に観察することで、画像処理等によって最適条件のマイクロリアクタ10を容易に検出することもできる。

【0018】次に、図3を参照してマイクロリアクタの製造工程を説明する。直径4インチ、厚さ0.7mmのシリコン基板11を用意し、表面を酸化してエッチングマスクを形成した後、フォトレジストを表面に塗布して、チャネルのパターンに合わせて露光/現像処理をした。そして、水酸化カリウム若しくはエチレンジアミン水溶液をエッチング液とした結晶方位依存性エッチング（異方性エッチング）によって、チャネル幅70μm、深さ1μmのチャネル14を形成した（図3（A））。異方性エッチングにより、アスペクト比の高い溝（チャネル）を形成することができる。

【0019】次いで、注入ポート12、13が取り付けられるべき位置に合わせて、集束イオンビーム加工（F

1 B加工)を利用してチャネル14に貫通孔16を穿設した(同図(B))。同図に図示していないが、排出ポート15に連通する貫通孔も同時に穿設した。平板17として、耐熱ガラス(商標名:バイレックス)を用い、350℃~400℃に加熱した状態でシリコン基板11と平板17間に500V~1000Vの電圧を印加して陽極接合をした(同図(C))。

【0020】最後に、貫通孔16の開孔位置に合わせて注入ポート12、13を取りつければ、マイクロリアクタ10が完成する(同図(D))。注入ポート12、13は、振動板19を取りつけた圧電体素子18を耐溶剤性のエポキシ系接着剤エコボンド(日本エイブルスティック社製)を用いてシリコン基板11に接着することで形成できる。この接着剤が硬化することで、側壁20が形成される。また、同図に図示していないが、排出ポート15についても同様に取り付け。さらに、半導体レーザー22、光センサ23、及び計測/制御部24等の各種計測機器をマイクロリアクタ10に取り付ければ、所望の化学反応実験に用いることができる。

【0021】尚、注入ポート12、13への試料の注入法として、例えば、図4に示すように、タンク32内に充填した試料40をシリコンチューブ31を介してキャビティ21内へ供給するように構成してもよい。圧電体素子はマイクロポンプ、インクジェット式記録ヘッドのインク吐出駆動源等の電気機械変換素子として利用されているため、圧電体素子18の駆動力を利用することで、タンク32内に充填された試料40をチャネル14内へ供給することができる。

【0022】また、図5に示すように、注入ポート12、13を構成する振動板19をヒンジ25を介して側壁20に取り付けることで、振動板19を開閉可能に構成することもできる。このような構成により、振動板19を開いた状態で液体吐出ヘッド30を用いて試料40をキャビティ21に充填することができる(液剤配置)。液体吐出ヘッド30としては、例えば、インクジェットプリンタに用いられるインクジェット式記録ヘッドを利用することができる。当該ヘッドは圧電体素子の体積変化により所望の液体を吐出させるピエゾジェット方式であってもよく、また、熱の印加により急激に蒸気が発生することにより液体を吐出させるバブルジェット方式であってもよい。キャビティ21に試料40を充填した後、振動板19を閉じて圧電体素子18を駆動すれば、チャネル14内への試料40の送受液が可能になる。また、注入ポート12、13をシリコン基板11から脱着可能に構成し、液体吐出ヘッド30を用いてキャビティ21内へ直接試料40を吐出するように構成してもよい。

【0023】尚、上記の説明においては、圧電体素子18の駆動によりキャビティ21内の試料を送受液するピエゾジェット方式を例に説明したが、熱の印加により急

激に蒸気が発生することにより試料を送液するバブルジェット方式を採用してもよい。本方式による場合、抵抗ヒータを含んで構成されるサーマル・エネルギー・ジェネレータに電気パルスを与えることで、試料の液相/気相間の相転移を利用してキャビティ21内に充填された試料をチャネル14へ送液する。

(実施例)上記の構成において、注入ポート12にATP(アデノシン三リン酸)溶液を注入し、注入ポート13にルシフェリンとルシフェラーゼの混合溶液を注入してリアクタにおけるホタルルシフェラーゼ反応を光学的に観察した。ホタルルシフェラーゼ反応は、酵素ルシフェラーゼがルシフェリン及びATPを基質として反応が進行する発光反応で、ルシフェリンの酸化に伴って光が発せられるため、リアクタ部分での発光強度をモニターすることにより、反応のアクティビティを知ることができる。ATP溶液の濃度をa、ルシフェリンとルシフェラーゼの混合溶液の濃度をbとし、注入ポート12、13のそれぞれの送液量をドットあたり10 p l/secの割合に設定し、各注入ポート12、13での供給量(ドット数)を変化させ、反応における濃度を変化させたところ、発光強度Iは図6のようになった。また、計測/制御部24において、検出した発光強度に応じて注入ポート12の送液量を0.1 p l/secの割合に変更し、注入ポート13の送液量を20 p l/secの割合に変更したところ、図7のように発光強度が変化した。

【0024】以上の実験結果から、圧電体素子18の駆動周波数を制御することで、チャネル14への送受液量を微妙に調整し、所望の反応の進行を得る(反応を自在に操作)ことができることを確認できた。このように、チャネル14への送受液手段として圧電体素子18を用いることで、シリンジポンプ或いはダイアフラム型のマイクロポンプでは実現できなかった精度で送受液をすることができ、分子のハンドリングに優れたマイクロリアクタを提供することができる。特に、圧電体素子18を用いる場合は、圧電セラミックスの組成を選択することで多種類の駆動波形を得ることができるため、マイクロリアクタにおける送受液手段として好適である。

【0025】

【発明の効果】本発明によれば、注入ポートとチャネル間の送受液を精密な精度で実現するマイクロリアクタを提供することができる。また、注入ポートのキャビティ内への試料の注入を精密な精度で実現することができる。

【図面の簡単な説明】

【図1】マイクロリアクタの平面図である。

【図2】図1のA-A断面図である。

【図3】マイクロリアクタの製造工程断面図である。

【図4】注入ポートへの試料供給を説明する図である。

【図5】注入ポートへの試料供給を説明する図である。

【図6】ホタルルシフェラーゼ反応の実験結果である。

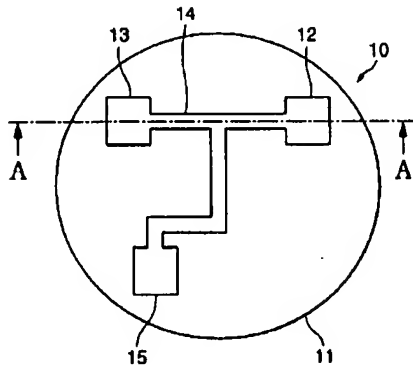
【図7】ホタルルシフェラーゼ反応の実験結果である。

【符号の説明】

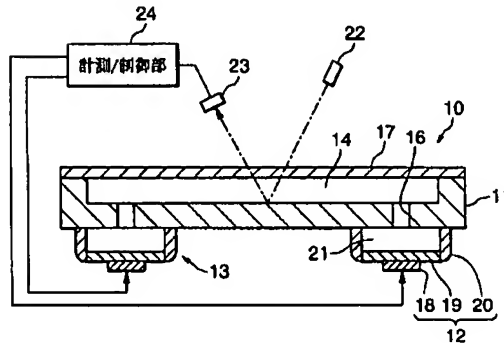
10…マイクロリアクタ、11…シリコン基板、12…  
注入ポート、13…注入ポート、14…チャンネル、15

…排出ポート、16…貫通孔、17…平板、18…圧電  
体素子、19…振動板、20…側壁、21…キャビテ  
ィ、22…半導体レーザ、23…光センサ、24…計測  
／制御部、25…ヒンジ、30…液体吐出ヘッド、31  
…シリコンチューブ、32…タンク、40…試料

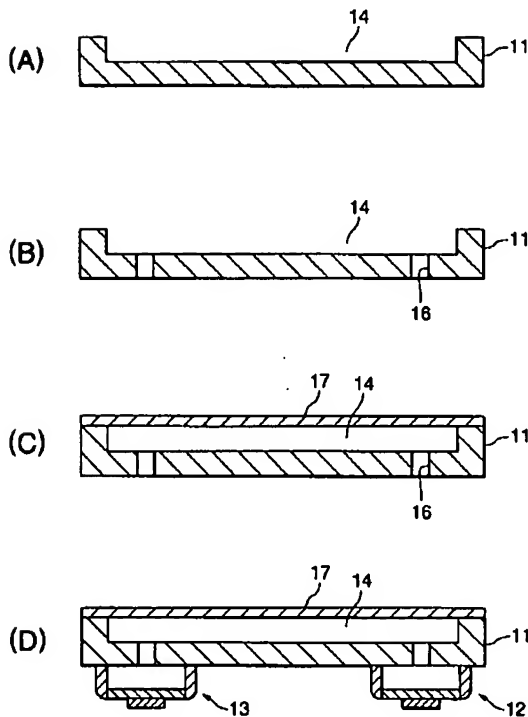
【図1】



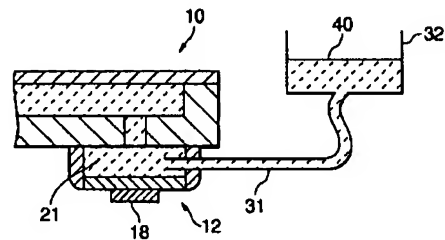
【図2】



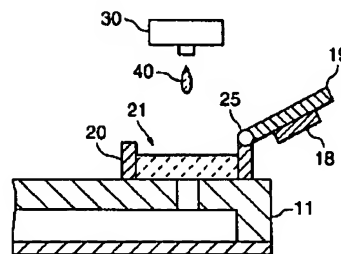
【図3】



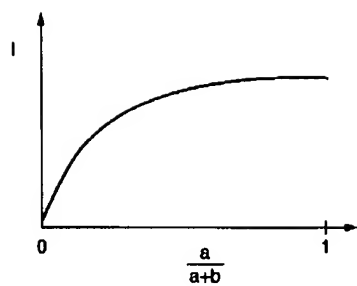
【図4】



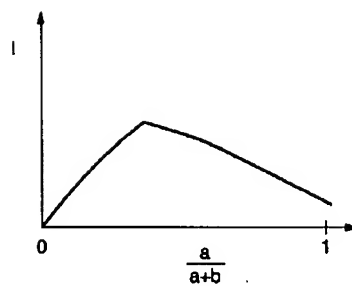
【図5】



【図6】



【図7】



フロントページの続き

Fターム(参考) 2G042 AA01 AA10 CB03 HA02 HA10  
 2G058 EA14 EB00 GA02 GA11 GB02  
 4G057 AB31 AB34  
 4G068 AA02 AA03 AA04 AA06 AA07  
 AB11 AB15 AC20 AF31  
 4G075 AA02 BA10 BD15 CA32 CA36  
 DA02 DA08 DA20 EB50 EC01  
 FC13



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CLAIMS

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[Claim(s)]

[Claim 1] The micro reactor equipped with the impregnation port which carries out the transmission-and-reception liquid of the sample between channels with an ink jet method, and the discharge port for it being open for free passage in an impregnation port through the channel concerned, and collecting the samples after a chemical reaction.

[Claim 2] The micro reactor according to claim 1 further equipped with the detector which detects the chemical reaction of a sample, and the control section which adjusts the transmission-and-reception volume of the sample from an impregnation port to a channel based on the detected data.

[Claim 3] Said detector is a micro reactor according to claim 2 which detects the chemical reaction of a sample optically.

[Claim 4] The micro reactor equipped with the impregnation port which the volume of a cavity is changed by the drive of a piezo electric crystal component, and carries out the transmission-and-reception liquid of the sample between channels, and the discharge port for it being open for free passage in an impregnation port through the channel concerned, and collecting the samples after a chemical reaction.

[Claim 5] The micro reactor according to claim 4 further equipped with the detector which detects the chemical reaction of a sample, and the control section which controls the drive of a piezo electric crystal component based on the detected data, and adjusts the transmission-and-reception volume of a sample.

[Claim 6] Said detector is a micro reactor according to claim 5 which detects the chemical reaction of a sample optically.

[Claim 7] Said impregnation port is a micro reactor given in any or the 1st term among claim 4 which introduces into a cavity the sample stored in the tank by the drive of a piezo electric crystal component, and sends the liquid to a channel further thru/or claim 6.

[Claim 8] Said piezo electric crystal component is a micro reactor given in any or the 1st term among claim 4 which carries out transmission-and-reception liquid between channels with the resolving power of 0.5pl(s) thru/or 60pl(s) thru/or claim 7.

[Claim 9] The sample restoration approach which fills up the cavity of the impregnation port of a micro reactor with a sample using a liquid discharge head.

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the amelioration technique of the micro reactor for performing a chemical reaction.

[0002]

[Description of the Prior Art] In order to perform many biochemical reactions to JP,10-337173,A in juxtaposition, the technique of using a micro reactor is proposed. The micro reactor is used for a chemical reaction experiment in a micro field, development of a chemical, artificial organ development, the genomic DNA analysis tool, the basic analysis tool of micro fluid engineering, etc. There is the description which is not in the usual chemical reaction which used the flask in the chemical reaction using this micro reactor. For example, since the whole equipment is small, effectiveness is very high, and there is an advantage which can perform temperature control efficiently. If the reaction which needs precise temperature control if this advantage is used, and the reaction which needs rapid heating or rapid cooling also use a micro reactor, it will become possible [ carrying out easily ].

[0003] Moreover, in order to react in minute space, in any case, compared with the volume of a liquid, the rate of the area of an interface is very large in the liquid and the liquid junction side of an organic solvent and water, and \*\* and the liquid junction side of a liquid and a container wall. So, the passing speed of a molecule is quick and a heterogeneous reaction can be performed efficiently. Furthermore, since the volume of a reactor (reaction vessel) is minute, the amount and cost of samples (a reaction reagent, sample, etc.) which are used for a reaction can be held down, and effect on an environment can be made small by making a reaction scale small to the limitation of the analysis capacity of a product.

[0004]

[Problem(s) to be Solved by the Invention] By the way, since the volume of a channel (passage) or a reactor is minute, a micro reactor requires handling of a different molecule from the usual reaction container. Although the sample was sent in the channel or the reactor using the syringe pump or the micropump of a diaphragm mold in the conventional micro reactor, in the micro reactor with a minute and complicated channel, exact liquid-sending control was difficult by the above-mentioned sending-the liquid method (pressure pumping).

[0005] For this reason, although the sending-the liquid method (electroosmotic pumping) by the electroosmotic method is used, it is unsuitable for a difficult organic solvent to form an electric double layer in an interface by this approach. H. The quality of electric field is added to an organic solvent, and the experimental result using an electroosmotic method is reported to SalimiMoosavi, T.Tang, J.Am.Chem.Soc, and 119 and 8716 (1997). Thus, in the micro reactor, it has been an important technical problem to realize handling of a precise molecule.

[0006] Then, this invention makes it a technical problem to offer a micro reactor equipped with the impregnation port which made possible the transmission-and-reception liquid to a channel in a precise precision. Moreover, let it be a technical problem to offer the sample restoration approach which fills up impregnation of the sample into the cavity of an impregnation port with a precise precision.

[0007]

[Means for Solving the Problem] The micro reactor of this invention is equipped with the impregnation port which carries out the transmission-and-reception liquid of the sample between channels, and the discharge port for it being open for free passage in an impregnation port through the channel concerned, and collecting the samples after a chemical reaction, and is constituted by the ink jet method in order to solve the above-mentioned technical problem. You may be the piezo jet method which the volume of the cavity formed in the wall of an impregnation port of mechanical displacement of for example, a piezo electric crystal component is changed to the transmission-and-reception liquid of the sample by the ink jet method, and sends the sample in a cavity, and when a steam occurs rapidly by impression of heat, you may be the Bubble Jet which sends a sample.

[0008] If it is the piezo jet method which makes a piezo electric crystal component a driving source, since transmission-and-reception liquid can be carried out between channels with the resolving power of 0.5pl thru/or 60pl(s), the transmission-and-reception liquid between an impregnation port and a channel is realizable in a precise precision.

[0009] In the above-mentioned configuration, you may have the detector which detects the chemical reaction of a sample, and the control section which adjusts the transmission-and-reception volume from an impregnation port to a channel based on the detected data further. Thus, with constituting, feedback control of the transmission-and-reception volume can be carried out according to the condition of the chemical reaction of a sample. In this case, a means to detect optically is suitable as a detection means of a chemical reaction. It is because it excels in detection in a micro field.

[0010] As a configuration for supplying a sample into the cavity of an impregnation port, the sample stored in the tank may be introduced into a cavity by the drive of a piezo electric crystal component, and you may constitute so that the liquid may be further sent to a channel.

[0011] Moreover, a sample is filled up with the sample restoration approach of this invention into the cavity of the impregnation port of a micro reactor using a liquid discharge head.

[0012]

[Embodiment of the Invention] Hereafter, the gestalt of this operation is explained with reference to each drawing.

[0013] The typical conceptual diagram of the micro reactor 10 is shown in drawing 1 . The micro reactor 10 can be manufactured by carrying out micro processing of the silicon substrate 11, is equipped with the impregnation ports 12 and 13, a channel 14, and the discharge port 15, and is constituted. A channel 14 is passage which opens the impregnation ports 12 and 13 and the discharge port 15 for free passage mutually, and constitutes a T character mold reactor. The sample poured into the impregnation ports 12 and 13 joins through a channel 14, and the downstream of a channel 14 functions as a reactor from the unification part concerned. Hereafter, the part which the chemical reaction has produced among channels 14 is called reactor. The samples after a reaction are collected from the discharge port 15. In addition, in order to carry out simple [ of the explanation ], in this drawing, the semiconductor laser 22 and photosensor 23 which are mentioned later, and the measurement / control-section 24 grade are not illustrating.

[0014] Drawing 2 is the A-A sectional view of drawing 1 . The plate 17 was stuck on the channel 14 formed in the front face of a silicon substrate 11, and the channel 14 is sealed. Moreover, the through tube (orifice) 16 is carrying out opening to the location corresponding to each port so that a sample can be supplied into a channel 14 from the impregnation ports 12 and 13. The impregnation ports 12 and 13 consist of a piezo electric crystal component 18, a diaphragm 19, and a side attachment wall 20. The cavity 21 for being filled up with a sample is formed of the wall and silicon substrate 11 of the impregnation ports 12 and 13. The piezo electric crystal component 18 is a component which transforms electrical energy into mechanical energy, and is equipped with the piezoelectric ceramics displaced according to an inverse piezoelectric effect, for example, PZT, (PZT). If an electrical potential difference is impressed to this electrostrictive ceramics, the piezo electric crystal component 18 will be contracted crosswise while it expands in the direction of thickness. Compressive stress works to the interface of the piezo electric crystal component 18 and a diaphragm 19, and the volume of a cavity 21

changes with these contraction. Consequently, liquid sending of the sample in a cavity 21 is attained to a channel 14 through a through tube 16. Since the piezo electric crystal component 18 can be driven with the oscillation frequency of several MHz, it is suitable for precise transmission-and-reception liquid control. The diaphragm 19 consists of insulator layers, such as silicon oxide and a silicon nitride.

[0015] The plate 17 consists of transparence substrates like heat-resisting glass, and can detect the chemical reaction in a reactor optically. Spectroscopy can be used as an optical detection means of the chemical reaction in a reactor. In this drawing, the incident light injected from semiconductor laser 22 penetrates the resultant in a plate 17 and a reactor, and after reflecting on the wall surface of a reactor, it is detected by the photosensor 23. Measurement / control section 24 measures the reinforcement of incident light, and the reinforcement of the reflected light, and carries out the quantum of the amount of resultants in a reactor. Measurement / control section 24 is equipped with the drive control circuit of the piezo electric crystal component 18 in the impregnation ports 12 and 13, and the transmission-and-reception volume of the sample in the impregnation ports 12 and 13 is constituted in the range of 0.5pl-60pl possible [ adjustment ] with reference to the amount of resultants in a reactor.

[0016] In addition, the laser fluorescence detector other than the above may be used as the detection approach of the chemical reaction in a reactor. Laser can be efficiently condensed by using an objective lens, and can detect high sensitivity. Moreover, a laser beam is irradiated all over a micro reactor, and the situation of impregnation of a sample, a chemical reaction, and separation can also be photoed with a high sensitivity camera. Although only analysis of the sample in an outlet can be performed in an electrochemical detector or a mass spectroscope, the phenomenon of the whole micro reactor is observable by such method. Moreover, it is effective, if it uses Hadamard transform electrophoresis analysis together in using a fluorescence method and an extinction method for detection of a sample.

[0017] Moreover, many micro reactors 10 from which a reaction condition differs can be arranged, and an image processing etc. can also detect the micro reactor 10 of optimum conditions easily by observing a reaction within many reactors to coincidence using a high-speed film or a CCD camera.

[0018] Next, the production process of a micro reactor is explained with reference to drawing 3 . After having prepared the silicon substrate 11 with a diameter [ of 4 inches ], and a thickness of 0.7mm, oxidizing the front face and forming an etching mask, the photoresist was applied to the front face and exposure/development was carried out according to the pattern of a channel. And the channel 14 with a channel width [ of 70 micrometers ] and a depth of 1 micrometer was formed by crystal orientation dependency etching (anisotropic etching) which used the potassium hydroxide or the ethylenediamine water solution as the etching reagent ( drawing 3 (A)). By anisotropic etching, the high slot on the aspect ratio (channel) can be formed.

[0019] Subsequently, according to the location in which the impregnation ports 12 and 13 should be attached, the through tube 16 was drilled in the channel 14 using focusing ion beam machining (FIB processing) (this drawing (B)). The through tube which is illustrating to this drawing and is open for free passage in \*\*\*\* and the discharge port 15 was also drilled in coincidence. The electrical potential difference of 500V-1000V was impressed between the silicon substrate 11 and the plate 17 in the condition of having heated at 350 degrees C - 400 degrees C, using heat-resisting glass (brand name: Pyrex) as a plate 17, and anode plate junction was carried out (this drawing (C)).

[0020] Finally, if the impregnation ports 12 and 13 are attached according to the opening location of a through tube 16, the micro reactor 10 will be completed (this drawing (D)). The impregnation ports 12 and 13 can form the piezo electric crystal component 18 which attached the diaphragm 19 by pasting a silicon substrate 11 using epoxy system adhesives Eko Bond (the Japanese Able stick company make) of solvent resistance. A side attachment wall 20 is formed because these adhesives harden. Moreover, although not illustrated to this drawing, it attaches similarly about the discharge port 15. Furthermore, if semiconductor laser 22, a photosensor 23, and the various measuring machine machines of measurement / control-section 24 grade are attached in the micro reactor 10, it can use for a desired chemical reaction experiment.

[0021] As a method of pouring in the sample to the impregnation ports 12 and 13, in addition, for example <A To HREF="/Tokujitu/tjitemdrw.ipdl?N0000=239&N0500=1

E\_N/;><==7>:6///&N0001=535&N0552=9&N 0553= 000006" TARGET="tjitemdrw"> drawing 4 You may constitute so that it may be shown, and the sample 40 with which it was filled up in the tank 32 may be supplied into a cavity 21 through the silicon tube 31. Since the piezo electric crystal component is used as electric machine sensing elements, such as an ink regurgitation driving source of a micropump and an ink jet type recording head, it can supply the sample 40 filled up with using the driving force of the piezo electric crystal component 18 in the tank 32 into a channel 14.

[0022] Moreover, as shown in drawing 5, it can also constitute from attaching in a side attachment wall 20 the diaphragm 19 which constitutes the impregnation ports 12 and 13 through a hinge 25 possible [ closing motion of a diaphragm 19 ]. By such configuration, where a diaphragm 19 is opened, a cavity 21 can be filled up with a sample 40 using the liquid discharge head 30 (liquids-and-solutions arrangement). As a liquid discharge head 30, the ink jet type recording head used for an ink jet printer can be used, for example. The head concerned may be a piezo jet method which makes a desired liquid breathe out by the volume change of a piezo electric crystal component, and when a steam occurs rapidly by impression of heat, it may be Bubble Jet which makes a liquid breathe out. If a diaphragm 19 is closed and the piezo electric crystal component 18 is driven after filling up a cavity 21 with a sample 40, the transmission-and-reception liquid of the sample 40 into a channel 14 will become possible. Moreover, the impregnation ports 12 and 13 may be constituted from a silicon substrate 11 possible [ desorption ], and you may constitute so that the regurgitation of the direct sample 40 may be carried out into a cavity 21 using the liquid discharge head 30.

[0023] In addition, in the above-mentioned explanation, although the piezo jet method which carries out the transmission-and-reception liquid of the sample in a cavity 21 by the drive of the piezo electric crystal component 18 was explained to the example, when a steam occurs rapidly by impression of heat, the Bubble Jet which sends a sample may be adopted. When based on this method, the sample filled up with giving an electric pulse to the thermal energy generator constituted including a resistance heater in the cavity 21 using the phase transition between the liquid phase/gaseous phase of a sample is sent to a channel 14.

(Example) In the above-mentioned configuration, the ATP (adenosine triphosphate) solution was poured into the impregnation port 12, the mixed solution of luciferin and luciferase was poured into the impregnation port 13, and the firefly luciferase reaction in a reactor was observed optically. A firefly luciferase reaction is a luminous reaction to which enzyme luciferase makes luciferin and ATP a substrate, and a reaction advances, and since light is emitted with oxidation of luciferin, it can know the activity of a reaction by carrying out the monitor of the luminescence reinforcement in a reactor part. When set concentration of the mixed solution of a, luciferin, and luciferase to b for the concentration of an ATP solution, each amount of liquid sending of the impregnation ports 12 and 13 was set as the rate of 10 pl/sec per dot, the amount of supply (the number of dots) in each impregnation ports 12 and 13 was changed and the concentration in a reaction was changed, the luminescence reinforcement I became like drawing 6. Moreover, in measurement / control section 24, when the amount of liquid sending of the impregnation port 12 was changed into the rate of 0.1 pl/sec according to the detected luminescence reinforcement and the amount of liquid sending of the impregnation port 13 was changed into the rate of 20 pl/sec, luminescence reinforcement changed like drawing 7.

[0024] It has checked that the transmission-and-reception volume to a channel 14 could be adjusted delicately, and advance of a desired reaction could be obtained from the above experimental result by controlling the drive frequency of the piezo electric crystal component 18 (a reaction is operated free). Thus, by using the piezo electric crystal component 18 as a transmission-and-reception liquid means to a channel 14, at a syringe pump or the micropump of a diaphragm mold, transmission-and-reception liquid can be carried out in the precision which was not able to be realized, and the micro reactor excellent in handling of a molecule can be offered. Since the drive wave of varieties can be acquired by choosing the presentation of electrostrictive ceramics especially when using the piezo electric crystal component 18, it is suitable as a transmission-and-reception liquid means in a micro reactor.

[0025]

[Effect of the Invention] According to this invention, the micro reactor which realizes the transmission-

and-reception liquid between an impregnation port and a channel in a precise precision can be offered. Moreover, impregnation of the sample into the cavity of an impregnation port is realizable in a precise precision.

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TECHNICAL FIELD

---

[Field of the Invention] This invention relates to the amelioration technique of the micro reactor for performing a chemical reaction.

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PRIOR ART

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[0003] Moreover, in order to react in minute space, in any case, compared with the volume of a liquid, the rate of the area of an interface is very large in the liquid and the liquid junction side of an organic solvent and water, and \*\* and the liquid junction side of a liquid and a container wall. So, the passing speed of a molecule is quick and a heterogeneous reaction can be performed efficiently. Furthermore, since the volume of a reactor (reaction vessel) is minute, the amount and cost of samples (a reaction reagent, sample, etc.) which are used for a reaction can be held down, and effect on an environment can be made small by making a reaction scale small to the limitation of the analysis capacity of a product.

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EFFECT OF THE INVENTION

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[Effect of the Invention] According to this invention, the micro reactor which realizes the transmission-and-reception liquid between an impregnation port and a channel in a precise precision can be offered. Moreover, impregnation of the sample into the cavity of an impregnation port is realizable in a precise precision.

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TECHNICAL PROBLEM

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[0005] For this reason, although the sending-the liquid method (electroosmotic pumping) by the electroosmotic method is used, it is unsuitable for a difficult organic solvent to form an electric double layer in an interface by this approach. H. The quality of electric field is added to an organic solvent, and the experimental result using an electroosmotic method is reported to SalimiMoosavi, T.Tang, J.Am.Chem.Soc, and 119 and 8716 (1997). Thus, in the micro reactor, it has been an important technical problem to realize handling of a precise molecule.

[0006] Then, this invention makes it a technical problem to offer a micro reactor equipped with the impregnation port which made possible the transmission-and-reception liquid to a channel in a precise precision. Moreover, let it be a technical problem to offer the sample restoration approach which fills up impregnation of the sample into the cavity of an impregnation port with a precise precision.

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MEANS

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[Means for Solving the Problem] The micro reactor of this invention is equipped with the impregnation port which carries out the transmission-and-reception liquid of the sample between channels, and the discharge port for it being open for free passage in an impregnation port through the channel concerned, and collecting the samples after a chemical reaction, and is constituted by the ink jet method in order to solve the above-mentioned technical problem. You may be the piezo jet method which the volume of the cavity formed in the wall of an impregnation port of mechanical displacement of for example, a piezo electric crystal component is changed to the transmission-and-reception liquid of the sample by the ink jet method, and sends the sample in a cavity, and when a steam occurs rapidly by impression of heat, you may be the Bubble Jet which sends a sample.

[0008] If it is the piezo jet method which makes a piezo electric crystal component a driving source, since transmission-and-reception liquid can be carried out between channels with the resolving power of 0.5pl thru/or 60pl(s), the transmission-and-reception liquid between an impregnation port and a channel is realizable in a precise precision.

[0009] In the above-mentioned configuration, you may have the detector which detects the chemical reaction of a sample, and the control section which adjusts the transmission-and-reception volume from an impregnation port to a channel based on the detected data further. Thus, with constituting, feedback control of the transmission-and-reception volume can be carried out according to the condition of the chemical reaction of a sample. In this case, a means to detect optically is suitable as a detection means of a chemical reaction. It is because it excels in detection in a micro field.

[0010] As a configuration for supplying a sample into the cavity of an impregnation port, the sample stored in the tank may be introduced into a cavity by the drive of a piezo electric crystal component, and you may constitute so that the liquid may be further sent to a channel.

[0011] Moreover, a sample is filled up with the sample restoration approach of this invention into the cavity of the impregnation port of a micro reactor using a liquid discharge head.

[0012]

[Embodiment of the Invention] Hereafter, the gestalt of this operation is explained with reference to each drawing.

[0013] The typical conceptual diagram of the micro reactor 10 is shown in drawing 1 . The micro reactor 10 can be manufactured by carrying out micro processing of the silicon substrate 11, is equipped with the impregnation ports 12 and 13, a channel 14, and the discharge port 15, and is constituted. A channel 14 is passage which opens the impregnation ports 12 and 13 and the discharge port 15 for free passage mutually, and constitutes a T character mold reactor. The sample poured into the impregnation ports 12 and 13 joins through a channel 14, and the downstream of a channel 14 functions as a reactor from the unification part concerned. Hereafter, the part which the chemical reaction has produced among channels 14 is called reactor. The samples after a reaction are collected from the discharge port 15. In addition, in order to carry out simple [ of the explanation ], in this drawing, the semiconductor laser 22 and photosensor 23 which are mentioned later, and the measurement / control-section 24 grade are not illustrating.

[0014] Drawing 2 is the A-A sectional view of drawing 1. The plate 17 was stuck on the channel 14 formed in the front face of a silicon substrate 11, and the channel 14 is sealed. Moreover, the through tube (orifice) 16 is carrying out opening to the location corresponding to each port so that a sample can be supplied into a channel 14 from the impregnation ports 12 and 13. The impregnation ports 12 and 13 consist of a piezo electric crystal component 18, a diaphragm 19, and a side attachment wall 20. The cavity 21 for being filled up with a sample is formed of the wall and silicon substrate 11 of the impregnation ports 12 and 13. The piezo electric crystal component 18 is a component which transforms electrical energy into mechanical energy, and is equipped with the piezoelectric ceramics displaced according to an inverse piezoelectric effect, for example, PZT, (PZT). If an electrical potential difference is impressed to this electrostrictive ceramics, the piezo electric crystal component 18 will be contracted crosswise while it expands in the direction of thickness. Compressive stress works to the interface of the piezo electric crystal component 18 and a diaphragm 19, and the volume of a cavity 21 changes with these contraction. Consequently, liquid sending of the sample in a cavity 21 is attained to a channel 14 through a through tube 16. Since the piezo electric crystal component 18 can be driven with the oscillation frequency of several MHz, it is suitable for precise transmission-and-reception liquid control. The diaphragm 19 consists of insulator layers, such as silicon oxide and a silicon nitride.

[0015] The plate 17 consists of transparence substrates like heat-resisting glass, and can detect the chemical reaction in a reactor optically. Spectroscopy can be used as an optical detection means of the chemical reaction in a reactor. In this drawing, the incident light injected from semiconductor laser 22 penetrates the resultant in a plate 17 and a reactor, and after reflecting on the wall surface of a reactor, it is detected by the photosensor 23. Measurement / control section 24 measures the reinforcement of incident light, and the reinforcement of the reflected light, and carries out the quantum of the amount of resultants in a reactor. Measurement / control section 24 is equipped with the drive control circuit of the piezo electric crystal component 18 in the impregnation ports 12 and 13, and the transmission-and-reception volume of the sample in the impregnation ports 12 and 13 is constituted in the range of 0.5pl-60pl possible [ adjustment ] with reference to the amount of resultants in a reactor.

[0016] In addition, the laser fluorescence detector other than the above may be used as the detection approach of the chemical reaction in a reactor. Laser can be efficiently condensed by using an objective lens, and can detect high sensitivity. Moreover, a laser beam is irradiated all over a micro reactor, and the situation of impregnation of a sample, a chemical reaction, and separation can also be photoed with a high sensitivity camera. Although only analysis of the sample in an outlet can be performed in an electrochemical detector or a mass spectroscope, the phenomenon of the whole micro reactor is observable by such method. Moreover, it is effective, if it uses Hadamard transform electrophoresis analysis together in using a fluorescence method and an extinction method for detection of a sample.

[0017] Moreover, many micro reactors 10 from which a reaction condition differs can be arranged, and an image processing etc. can also detect the micro reactor 10 of optimum conditions easily by observing a reaction within many reactors to coincidence using a high-speed film or a CCD camera.

[0018] Next, the production process of a micro reactor is explained with reference to drawing 3. After having prepared the silicon substrate 11 with a diameter [ of 4 inches ], and a thickness of 0.7mm, oxidizing the front face and forming an etching mask, the photoresist was applied to the front face and exposure/development was carried out according to the pattern of a channel. And the channel 14 with a channel width [ of 70 micrometers ] and a depth of 1 micrometer was formed by crystal orientation dependency etching (anisotropic etching) which used the potassium hydroxide or the ethylenediamine water solution as the etching reagent ( drawing 3 (A)). By anisotropic etching, the high slot on the aspect ratio (channel) can be formed.

[0019] Subsequently, according to the location in which the impregnation ports 12 and 13 should be attached, the through tube 16 was drilled in the channel 14 using focusing ion beam machining (FIB processing) (this drawing (B)). The through tube which is illustrating to this drawing and is open for free passage in \*\*\*\* and the discharge port 15 was also drilled in coincidence. The electrical potential difference of 500V-1000V was impressed between the silicon substrate 11 and the plate 17 in the condition of having heated at 350 degrees C - 400 degrees C, using heat-resisting glass (brand name:

Pyrex) as a plate 17, and anode plate junction was carried out (this drawing (C)).

[0020] Finally, if the impregnation ports 12 and 13 are attached according to the opening location of a through tube 16, the micro reactor 10 will be completed (this drawing (D)). The impregnation ports 12 and 13 can form the piezo electric crystal component 18 which attached the diaphragm 19 by pasting a silicon substrate 11 using epoxy system adhesives Eko Bond (the Japanese Able stick company make) of solvent resistance. A side attachment wall 20 is formed because these adhesives harden. Moreover, although not illustrated to this drawing, it attaches similarly about the discharge port 15. Furthermore, if semiconductor laser 22, a photosensor 23, and the various measuring machine machines of measurement / control-section 24 grade are attached in the micro reactor 10, it can use for a desired chemical reaction experiment.

[0021] In addition, as a method of pouring in the sample to the impregnation ports 12 and 13, as shown in drawing 4 , you may constitute so that the sample 40 with which it was filled up in the tank 32 may be supplied into a cavity 21 through the silicon tube 31. Since the piezo electric crystal component is used as electric machine sensing elements, such as an ink regurgitation driving source of a micropump and an ink jet type recording head, it can supply the sample 40 filled up with using the driving force of the piezo electric crystal component 18 in the tank 32 into a channel 14.

[0022] Moreover, as shown in drawing 5 , it can also constitute from attaching in a side attachment wall 20 the diaphragm 19 which constitutes the impregnation ports 12 and 13 through a hinge 25 possible [ closing motion of a diaphragm 19 ]. By such configuration, where a diaphragm 19 is opened, a cavity 21 can be filled up with a sample 40 using the liquid discharge head 30 (liquids-and-solutions arrangement). As a liquid discharge head 30, the ink jet type recording head used for an ink jet printer can be used, for example. The head concerned may be a piezo jet method which makes a desired liquid breathe out by the volume change of a piezo electric crystal component, and when a steam occurs rapidly by impression of heat, it may be Bubble Jet which makes a liquid breathe out. If a diaphragm 19 is closed and the piezo electric crystal component 18 is driven after filling up a cavity 21 with a sample 40, the transmission-and-reception liquid of the sample 40 into a channel 14 will become possible. Moreover, the impregnation ports 12 and 13 may be constituted from a silicon substrate 11 possible [ desorption ], and you may constitute so that the regurgitation of the direct sample 40 may be carried out into a cavity 21 using the liquid discharge head 30.

[0023] In addition, in the above-mentioned explanation, although the piezo jet method which carries out the transmission-and-reception liquid of the sample in a cavity 21 by the drive of the piezo electric crystal component 18 was explained to the example, when a steam occurs rapidly by impression of heat, the Bubble Jet which sends a sample may be adopted. When based on this method, the sample filled up with giving an electric pulse to the thermal energy generator constituted including a resistance heater in the cavity 21 using the phase transition between the liquid phase/gaseous phase of a sample is sent to a channel 14.

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[Translation done.]

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- 3.In the drawings, any words are not translated.

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EXAMPLE

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(Example) In the above-mentioned configuration, the ATP (adenosine triphosphate) solution was poured into the impregnation port 12, the mixed solution of luciferin and luciferase was poured into the impregnation port 13, and the firefly luciferase reaction in a reactor was observed optically. A firefly luciferase reaction is a luminous reaction to which enzyme luciferase makes luciferin and ATP a substrate, and a reaction advances, and since light is emitted with oxidation of luciferin, it can know the activity of a reaction by carrying out the monitor of the luminescence reinforcement in a reactor part. When set concentration of the mixed solution of a, luciferin, and luciferase to b for the concentration of an ATP solution, each amount of liquid sending of the impregnation ports 12 and 13 was set as the rate of 10 pl/sec per dot, the amount of supply (the number of dots) in each impregnation ports 12 and 13 was changed and the concentration in a reaction was changed, the luminescence reinforcement I became like drawing 6 . Moreover, in measurement / control section 24, when the amount of liquid sending of the impregnation port 12 was changed into the rate of 0.1 pl/sec according to the detected luminescence reinforcement and the amount of liquid sending of the impregnation port 13 was changed into the rate of 20 pl/sec, luminescence reinforcement changed like drawing 7 .

[0024] It has checked that the transmission-and-reception volume to a channel 14 could be adjusted delicately, and advance of a desired reaction could be obtained from the above experimental result by controlling the drive frequency of the piezo electric crystal component 18 (a reaction is operated free). Thus, by using the piezo electric crystal component 18 as a transmission-and-reception liquid means to a channel 14, at a syringe pump or the micropump of a diaphragm mold, transmission-and-reception liquid can be carried out in the precision which was not able to be realized, and the micro reactor excellent in handling of a molecule can be offered. Since the drive wave of varieties can be acquired by choosing the presentation of electrostrictive ceramics especially when using the piezo electric crystal component 18, it is suitable as a transmission-and-reception liquid means in a micro reactor.

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[Translation done.]

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DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]

[Drawing 1] It is the top view of a micro reactor.

[Drawing 2] It is the A-A sectional view of drawing 1 .

[Drawing 3] It is the production process sectional view of a micro reactor.

[Drawing 4] It is drawing explaining sample supply in an impregnation port. .

[Drawing 5] It is drawing explaining sample supply in an impregnation port.

[Drawing 6] It is the experimental result of a firefly luciferase reaction.

[Drawing 7] It is the experimental result of a firefly luciferase reaction.

[Description of Notations]

a 10 -- micro reactor, 11 -- silicon substrate, and 12 -- an impregnation port, 13 -- impregnation port, 14 -- channels, and 15 -- a discharge port, 16 -- through tube, 17 -- plate, and 18 -- a piezo electric crystal component, 19 -- diaphragm, 20 -- side attachment wall, and 21 -- a cavity, 22 -- semiconductor laser, 23 -- photosensor, and 24 -- measurement/control section, 25 -- hinge, 30 -- liquid discharge head, and 31 -- a silicon tube, 32 -- tank, and 40 -- sample

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[Translation done.]

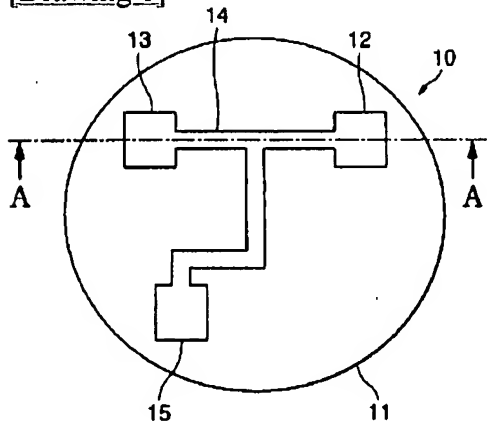
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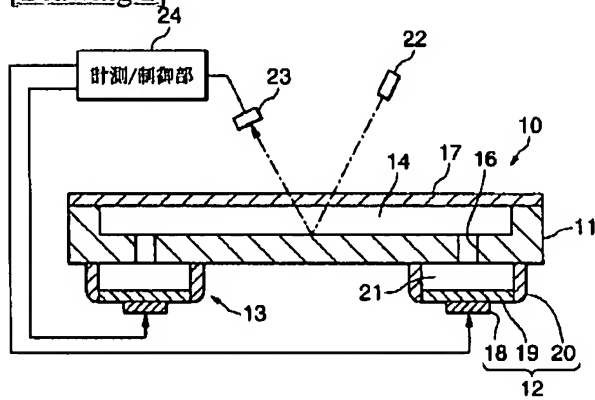
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## DRAWINGS

[Drawing 1]

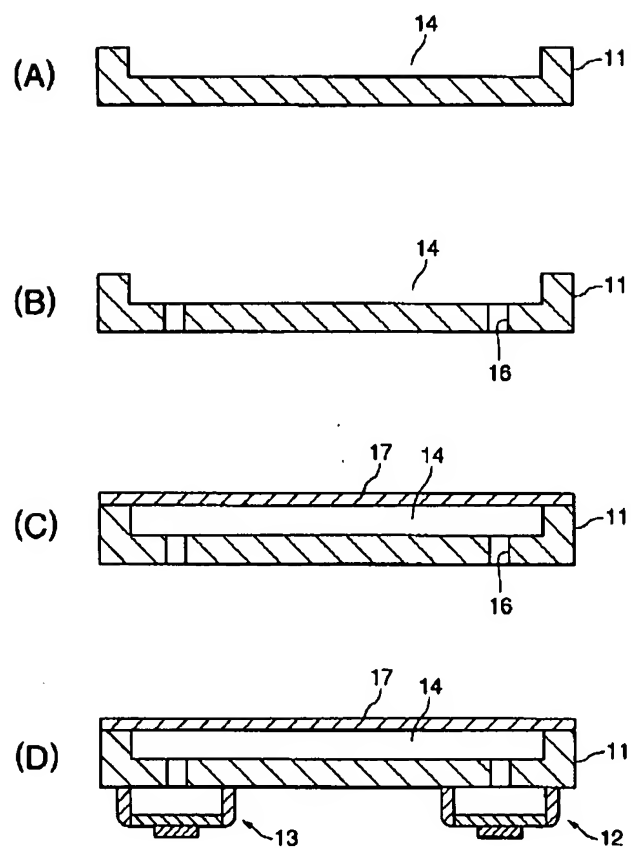


[Drawing 2]

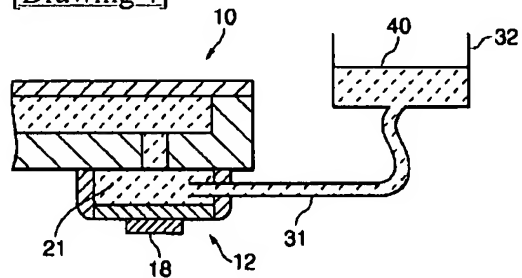


[Drawing 3]

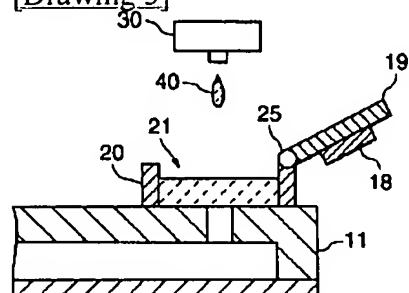




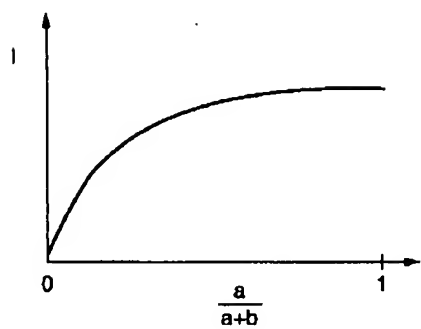
[Drawing 4]



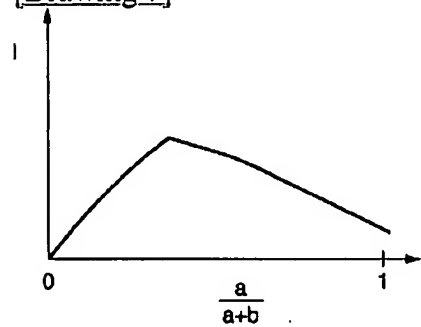
[Drawing 5]



[Drawing\_6]



[Drawing 7]



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[Translation done.]